

DETERMINATION OF GROUP COMPOSITION OF PETROLEUM FRACTIONS  
IN A LIQUID CHROMATOGRAPH

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(NASA-TT-F-16289) DETERMINATION OF GROUP  
COMPOSITION OF PETROLEUM FRACTIONS IN A  
LIQUID CHROMATOGRAPH (Scientific Translation  
Service) 6 p HC \$3.25 CSCI 07D

N75-22402

Unclass

G3/25 19495

Translation of: "Opredeleniye gruppobogo  
sostava neftyanykh fraktsiy na zhidkost-  
nom khromatografe," Neftepererabotka i  
Neftekhimiia (Moscow), No. 2, 1971,  
pp. 32 - 54.



1. Report No. NASA TT F-16,289		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle DETERMINATION OF GROUP COMPOSITION OF PETROLEUM FRACTIONS IN A LIQUID CHROMATOGRAPH				5. Report Date May 1975	
				6. Performing Organization Code	
7. Author(s) T. S. Ivanova, Yu. A. Burkin and V. G. Zizin				8. Performing Organization Report No.	
				10. Work Unit No.	
9. Performing Organization Name and Address SCITRAN Box 5456 Santa Barbara, CA 93108				11. Contract or Grant No. NASw-2483	
				13. Type of Report and Period Covered Translation	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Translation of "Opredeleniye gruppobogo sostava neftyanykh fraktsiy na zhidkostnom khromatografe," Neftepererabotka i Neftekhimiia (Moscow), No. 2, 1971, pp. 32 - 34.					
16. Abstract  A new prototype model of a liquid chromatograph with automatic recording of analysis results, built at the Bashkir Scientific Research Institute of the Petroleum Industry, is described.					
17. Key Words (Selected by Author(s))				18. Distribution Statement  Unclassified - Unlimited	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 6	
				22. Price	

DETERMINATION OF GROUP COMPOSITION OF PETROLEUM FRACTIONS  
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Adsorption liquid chromatography differs from the gas-liquid method in that its separating agent is a solid and the carrier some kind of solvent. This kind of chromatography at the present time is used for determining group hydrocarbon composition of petroleum kerosene-gas oil fractions primarily in research laboratories and is not widely used in control laboratories because it is very time consuming.

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At the present time a linear version of liquid-adsorption chromatography (LLAC)\* is being developed; the essence of the method is that active sorbents are modified by certain strong sorbents for the purpose of significantly increasing the linear section of the sorption isotherm. This version undoubtedly has a number of advantages in comparison with the usual method: first of all, it makes it possible to separate groups of hydrocarbons more clearly and with less loss of time in a shorter column; secondly, it allows automation of the process.

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Improved methods of separation, which increased its effectiveness, demanded the creation of new detecting instruments.

In order to obtain greater accuracy and sensitivity of the LLAC method a new prototype model of a liquid chromatograph with automatic recording of analysis results was designed and build by the Bashkir Scientific Research Institute of the Petroleum Industry.

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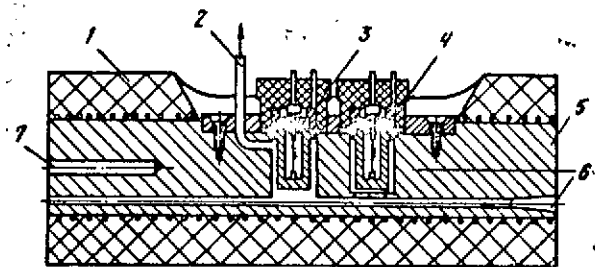
\*V. G. Zizin and T. S. Ivanova. "Zhurnal analiticheskoy khimi," No. 12, 1968.

\*\* Numbers in margin indicate pagination of original foreign text.

The liquid chromatograph with the automatic detecting instrument consists of three basic units: separation, detection and registration sections.

The sample passing from the column together with the carrier liquid is transported by a closed chain moving at a constant speed. The carrier liquid is removed by blowing. Catalytic combustion of the sample being analyzed occurs in platinum arm elements. Iso-octane is used as a carrier liquid; it is fed into the column at a rate of 0.25 ml/min.

A chromatographic column 1 m long with an inner diameter of 4 mm is filled with alumogel (150-200 mesh granulation); water in the amount of 3-3.5% of the weight of the alumogel is deposited on its surface. The amount of the sample to maintain a sample:adsorbent ratio of approximately 1:900 is 0.25 ml. Chromatography is conducted at room temperature. At the column outlet, the carrier liquid enters a continuously moving closed chain — approximately one drop per grain. The chain transports the eluate (mixture of carrier liquid and sample) in sequence through the carrier liquid evaporator and the thermochemical detector channel (see illustration). Air is drawn through the inlet and outlet of the preliminary evaporator at a rate of 10 l/min, exhausting the carrier liquid from the chain. The sample remaining in the chain is carried



Thermochemical detector of liquid chromatograph:

1 — detector furnace; 2 — pipe for drawing air through measuring arm element; 3 — measuring arm element; 4 — relative arm element; 5 — detector block; 6 — channel for transporter-chain; 7 — pocket for thermocouple.

into the thermochemical detector channel and heated by a furnace to a temperature of 150-170°C. The middle pipe of this channel is connected with the cell of the working arm element through which air is drawn off at a rate of 50 ml/min. A relative arm element is located inside the detector cell; it has a temperature identical with the working arm. The arm elements (working and relative) are included in the circuit of an alternating current automatic electronic balanced bridge and heated diagonally to approximately 600°C (with 0.1 mm cross section of the platinum the current in the detector must be 500 ma).

As it evaporates, the sample creates a concentration of hot substances in the air which burn on the platinum and raise the temperature of the working arm element.

The following conditions were chosen to separate hydrocarbon groups:

Rate of carrier liquid, ml/min	0.25
Amount of sample, ml	0.025-0.04
Rate of drawing air through evaporator, l/min	10.0
Temperature of sample evaporator, °C	150
Air dilution in detector, ml/min	50
Current of detector, ma	500

Sensitivity of the thermochemical detector under these conditions is approximately  $5 \cdot 10^{-7}$  ml/min.

Reproducibility of analysis results with this instrument was verified in an extract of aromatic hydrocarbons, extracted by dimethyl formamide from the 180-350°C fraction of Arlansk petroleum (Table 1).]

TABLE 1

## ANALYSIS OF EXTRACT OF AROMATIC HYDROCARBONS, % BY WEIGHT

Parallel determinations	Aromatic		
	saturated	monocyclic	bicyclic
1	24.10	35.80	40.10
2	23.10	34.80	42.10
3	23.30	35.00	41.70
4	23.70	34.00	42.70
Average	23.60	34.90	41.50
Relative deviation	±2.1	±2.6	+3.4 and -1.9

Table 2 gives data concerning analyses of artificial mixtures.

Thus, maximum error of the method does not exceed 10% rel. One determination of group composition takes 60-70 minutes.

TABLE 2

## ANALYSIS OF ARTIFICIAL MIXTURES, % BY WEIGHT

Results of parallel analyses	Aromatic		
	saturated	monocyclic	bicyclic
Taken	50.00	50.00	--
Obtained:			
1	46.00	54.00	--
2	47.7	52.30	--
Average	46.9	53.10	--
Relative error	-6.2	+6.2	--
Taken	0.5	--	99.5
Obtained:			
1	0.5	--	99.5
2	0.6	--	99.4
Average	0.55	--	99.45
Relative error	+10.0	--	-0.05
Taken	33.3	33.3	33.4
Obtained:			
1	35.4	30.2	34.4
2	36.6	29.8	33.6
Average	36.0	30.6	34.0
Relative error	+8.1	-10.0	+1.8